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New Technique for Measurement of Electron Diffraction Patterns of Polycrystalline Materials

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Prepared for COMMANDER SPACE SYSTEMS DIVISION
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Inglewood, California

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ABSTRACT

A technique ~~is described~~ for the measurement of electron diffraction patterns of polycrystalline materials. The image of the diffraction pattern is projected by a photographic enlarger at a predetermined magnification onto a chart that is calibrated directly in d spacings.

This technique is rapid, as no calculations are involved. Accuracy is equal to, or better than, that achieved with the classical method.

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I. INTRODUCTION

Electron diffraction has proven to be a valuable tool in the study of the structure of materials of small particle size, minute amounts of materials, and thin films. Even though it has the advantage of requiring only short exposure times (on the order of seconds), its use has been restricted by the time required to analyze the diffraction pattern. The classical method for measuring electron diffraction patterns utilizes a pattern of the unknown sample and of a known standard material, e.g., a gold film taken under identical conditions. The diameters (or radii) of the rings of both patterns are measured, using a divider and scale -- or, if available, a measuring microscope. The intensities of each of the rings are also recorded. A camera constant $(L\lambda)$[1] or conversion factor (K)[2] can be calculated from the standard pattern measurements, and from these computations the $\underline{d}$ values for the rings of the unknown pattern can be determined.

This method suffers from two limitations: (1) it is time consuming, as it involves the measurement of both the standard and the unknown patterns, and (2) two calculations are required -- that for the camera constant from the standard pattern, and that for the $\underline{d}$ values for the unknown.

In addition, the small size of the pattern makes it difficult to obtain accuracy to greater than two significant figures without the use of a measuring microscope.

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1 $dr = L\lambda$ or $d = \frac{L\lambda}{r}$ — d is the interplaner spacing, L = microscope focal length, $L\lambda$ is the camera constant for the microscope. (See Ref. 1 and 2)

2 $d = \frac{2f\lambda}{D} = \frac{K}{D}$ — d is the interplaner spacing, f = microscope focal length, K is a constant for a given apparatus and accelerating voltage. (See Ref. 3)

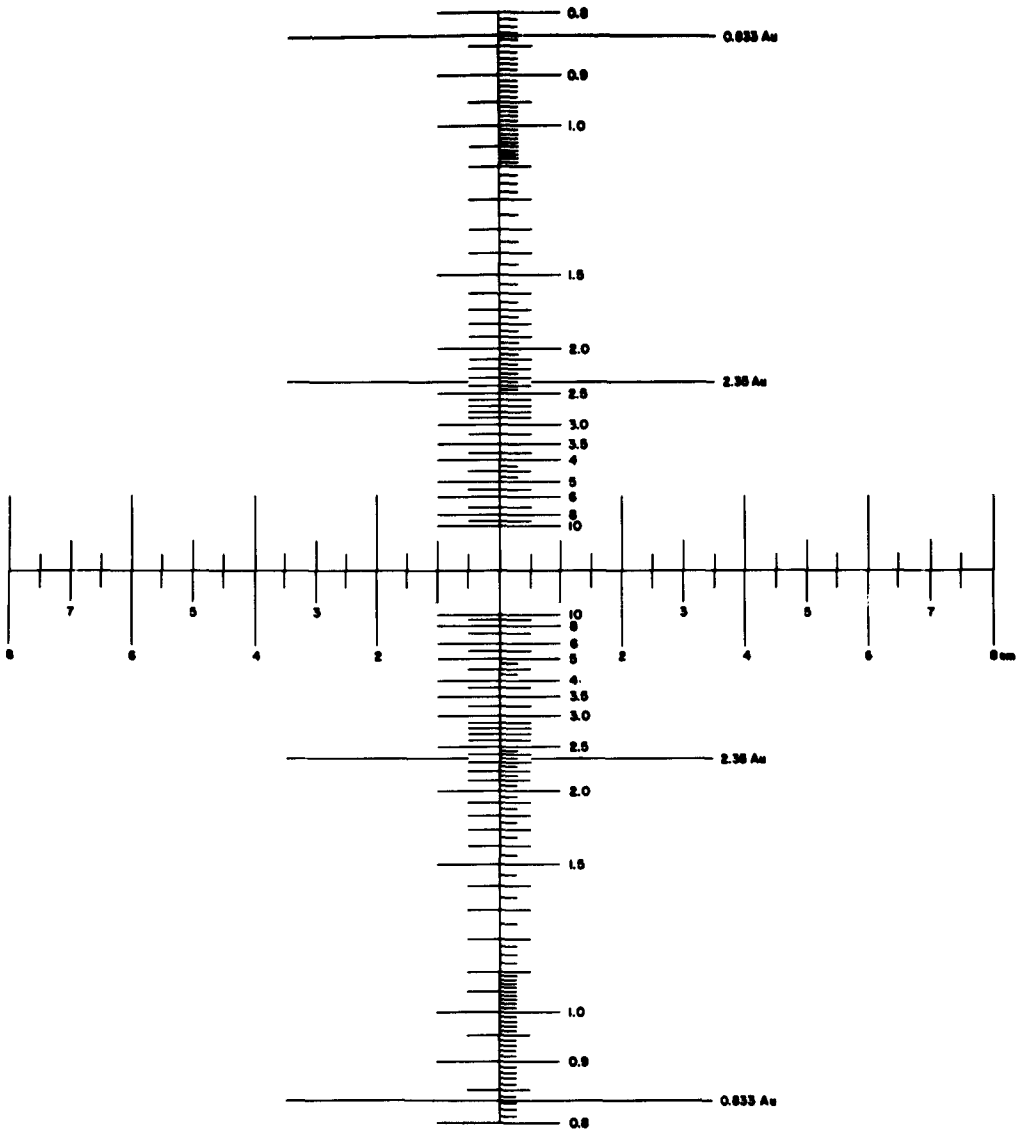

Fig. 1. Chart Used in the New Measurement Technique

II. NEW TECHNIQUE

In the new technique, the image of the diffraction pattern is projected by a photographic enlarger at a predetermined magnification onto a chart that is calibrated directly in $\underline{d}$ spacings. Separate calculations for each sample are eliminated in this manner. Furthermore, line measurements can be made with greater ease and accuracy because of the enlarged pattern. If the wavelength of the particular accelerating voltage to be used is substituted into the Bragg equation with appropriately chosen $\underline{d}$ values, the angle θ corresponding to each of these values can be calculated;[3] then a chart for the measurements of interplaner spacings may be constructed with these computed θ values. It may be necessary to multiply these θ angle values by some constant (c) to obtain a chart of the desired dimensions. The $\underline{d}$ values used in the calculations were chosen so that they fall within the range of the ASTM x-ray powder data file (0.80Å - 10.00Å) while giving a reasonable separation of lines on the chart when multiplied by (c). A sample computation of the distance (R) from the center of the chart to a given $\underline{d}$ spacing line (10Å) is given below.

$$\text{Sin } \theta = \frac{n\lambda}{2d} = \frac{0.037\text{Å}}{20\text{Å}} = 0.00185$$

$$\theta = 0.106 \text{ deg}$$

$$\theta \times c = 0.106 \times 70 = 7.42\text{mm} = R$$

c = chart size constant (mm/deg)

R = distance of 10Å line from center of chart

λ = 0.037Å (for 100 kV electrons)

[3] The sine of the angle θ was used rather than the approximation sin $\theta = \theta$ as in classical methods.

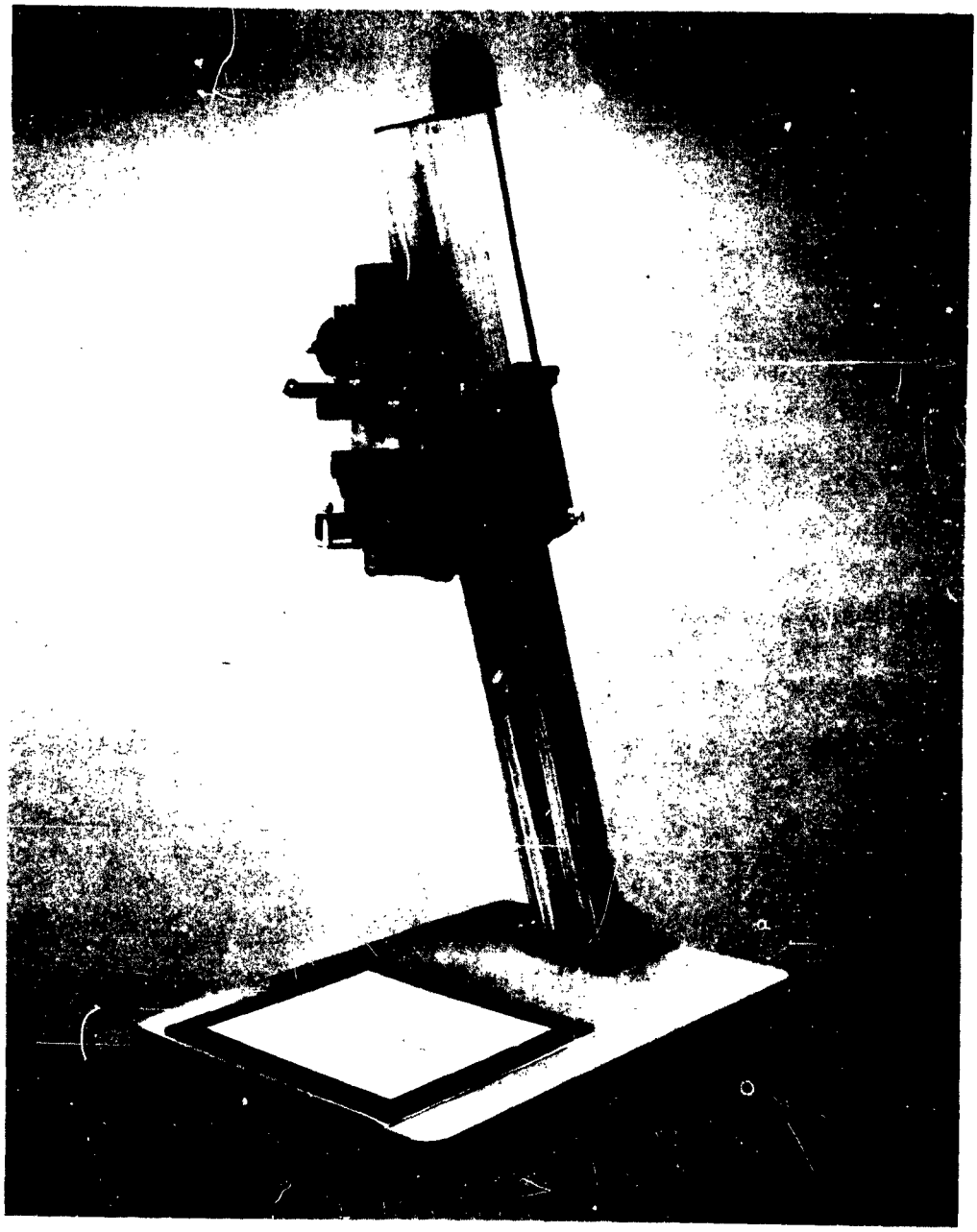

Fig. 2. Actual Laboratory Set-up Used in the New Measurement Technique

The angles subtended by the diffracted electrons are so small that the segment of the sphere on which they project very nearly coincides with a plane surface. Therefore, no correction was made for the projection of the diffraction pattern onto a planer, rather than a spherical, surface.

Figure 1 shows the chart used with patterns obtained on a Hitachi HU-11 electron microscope with an accelerating voltage of 100 kV. A value of (c) was chosen to give a chart size that matched a three-times enlargement of the electron diffraction pattern. The d value lines were ruled on both sides of the center line to facilitate centering of the chart with the electron diffraction pattern. Arbitrary centimeter rulings were also made at right angles to, and on both sides of, the d value lines for the same reason. The d values 2.355Å and 0.833Å are accentuated on the chart for calibration purposes.

When using the chart to measure an electron diffraction pattern, the arrangement shown in Figure 2 is used in a semidarkened room. Used with the chart is a 35 mm enlarger[4] with an appropriate lens.[5] An electron diffraction pattern of both the unknown and the gold standard are obtained under exactly the same conditions and on the same photographic plate, if possible. This plate is inserted in the enlarger, and the gold pattern is projected on the d value chart. The chart is aligned with the projected pattern by centering it, using the centimeter rulings in one direction and the d values in the other direction. The enlarger is raised or lowered until

[4] Omega Type A2, 35 mm Condenser Enlarger without plate or film holder.

[5] Schneider Componon f/5.6, 80 mm Enlarging Lens on modified lens board with plate-to-lens distance of approximately 80 mm. A shorter focal length lens yields severe spherical abberation of the projected diffraction pattern, whereas a longer focal length lens reduces intensity.

the 100 percent intensity line (2.355 Å) of the projected gold pattern corresponds roughly with the 2.355 Å line marked on the chart; it is important that the projected image be in focus. Then, with the chart kept exactly centered, the final calibration is accomplished by raising or lowering the enlarger and/or adjusting the focusing knob until the 0.833 Å lines on both sides of the chart centerline coincide exactly with the 0.833 Å line of the gold standard pattern. The photographic plate is shifted in the enlarger, the unknown pattern is projected, and the chart recentered under the pattern. The d-values of the lines are read directly from the chart and recorded along with the visually estimated intensities of the lines. These d values may be used with the ASTM X-Ray Powder Data File to characterize the unknown sample.

III. COMPARISON OF RESULTS

Measurements were made of a known material, magnesium, by both the classical and present methods; they are summarized in Table 1. can be seen from this Table that the deviations from the literature values are much less for the readings obtained by the present technique than for those obtained by the classical method. Missing lines in the electron diffraction pattern result from preferred orientation in the evaporated magnesium specimen.

In order to determine the degree of reproducibility of this method, three separate measurements were made of a standard pattern. The values obtained are listed in Table 2, along with the literature values. As can be seen from this Table, this method is quite reproducible, since the difference between any two runs, $\Delta L\mathring{A}$ is 0.01 $\mathring{A}$, or less. Numerous other materials, such as aluminum oxide and graphite, have been examined and have yielded equivalent results.

Table 1. Interplaner Distances Obtained by the New Measurement Technique and Classical Methods, Compared with those of the Literature

Magnesium, ASTM Powder Data File 4-0770		Transmission Pattern of Evaporated Magnesium Film					
		Classical Method (with divider and rule)			New Technique		
d(Å)	I/I_1	d(Å)	I/I_1	Deviation from Lit. Value (Å)	d(Å)	I/I_1	Deviation from Lit. Value (Å)
2.78	35	2.77	S	+0.01	2.78	S	0
2.61	41	---	---	---	---	---	---
2.45	100	2.44	VS	+0.01	2.45	VS	0
1.90	20	1.91	M	-0.01	1.90	M	0
1.605	18	1.59	M	+0.015	1.60	M	+0.005
1.473	18	1.50	W	-0.027	---	---	---
1.389	2	1.393	W	-0.004	1.39	W	-0.001
1.366	16	1.364	W+	+0.002	1.365	W+	+0.001
1.343	9	1.336	W	+0.007	1.34	W	+0.003
1.303	2	---	---	---	---	---	---
1.227	2	1.227	VW	0	1.225	VW	+0.002
1.180	2	---	---	---	---	---	---
1.085	2	1.093	VW	-0.008	1.085	VW	0
1.051	1	1.054	VW	-0.003	1.048	VW	+0.003
1.030	7	---	---	---	1.027	W	+0.003
1.011	3	1.019	VW	-0.008	---	---	---
0.976	2	0.982	VW	-0.006	0.970	VW	+0.006
0.951	1	---	---	---	---	---	---
0.927	1	0.930	VW	-0.003	0.923	VW	+0.004
0.899	4	0.909	VW	-0.010	0.900	VW	-0.001
0.873	2	0.880	VW	-0.007	0.870	VW	+0.003

Table 2. Reproducibility of the New Measurement Technique

Magnesium, ASTM Powder Data File 4-0770		Transmission Pattern of Evaporated Magnesium Film			Largest Difference any Two Runs
		Run No. 1	Run No. 2	Run No. 3	
d(Å)	I/I_1	d(Å)	d(Å)	d(Å)	ΔL(Å)
2.78	35	2.78	2.78	2.79	0.01
2.61	41	---	---	---	---
2.45	100	2.45	2.45	2.44	0.01
1.90	20	1.90	1.90	1.90	0.00
1.473	18	---	---	---	---
1.605	18	1.60	1.60	1.61	0.01
1.389	2	1.39	1.39	1.40	0.01
1.366	16	1.365	1.37	1.37	0.005
1.343	9	1.34	1.34	1.34	0.00
1.303	2	---	---	---	---
1.227	2	1.225	1.23	1.23	0.005
1.180	2	---	---	---	---
1.085	2	---	---	---	---
1.085	2	1.085	1.087	1.085	0.002
1.051	1	1.048	1.050	1.050	0.002
1.030	7	1.027	1.030	1.035	0.008
1.011	3	---	---	---	---
0.976	2	0.970	0.973	0.970	0.003
0.951	1	---	---	---	---
0.927	1	0.923	0.958	---	0.005
0.899	4	0.900	0.900	0.901	0.001
0.873	2	0.870	0.872	0.876	0.006

IV. CONCLUSION

This new technique has proven to be a fast and convenient method for measurement of electron diffraction patterns. Other advantages are that it eliminates the necessity for any calculation, it is more accurate than the classical method, and yet is very reproducible. In addition, it is inexpensive to set up: the only equipment required is an enlarger and the d-value chart.

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Aerospace Corporation, El Segundo, California. NEW TECHNIQUE FOR MEASUREMENT OF ELECTRON DIFFRACTION PATTERNS OF POLYCRYSTALLINE MATERIALS, prepared by J. H. Richardson and R. F. Schneidmiller, Materials Sciences Laboratory. 18 June 1963. [22] p. incl. illus. (Report TDR-169(3240-30)TN-1; SSD-TDR-63-83) (Contract AF 04(695)-169) Unclassified report

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